Environmental Health Perspectives
Vol. 9, pp. 57–61, 1974

Detection and Identification of Asbestos by Microscopical Dispersion Staining

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Asbestos fibers as small as 1 μm in diameter can be uniquely identified by light microscopy by employing dispersion staining methods. The technique described herein involves suspension of fibers in liquids of known refractive indices and observation of color display by means of a dispersion staining objective. Wavelengths or indices of refraction may be determined at right angles to and parallel to fiber axes. This method is rapid and sensitive for identification purposes.

There is a great need for a dependable, sensitive and rapid method for the detection and identification of asbestos. Microscopical dispersion staining satisfies all of these requirements. It is dependable because it is based on the measurement of three refractive indices as well as the dispersion of those indices. Refractive indices are among the most valuable identifying characteristics for small particles. The method is sensitive because the refractive indices are "read" as bright dispersion staining colors against a black background. These colors can be observed on particles well below 1 μm in diameter. It is rapid because any particle in the microscopical preparation showing the optical properties of asbestos signals its presence by a unique color combination with polarized light. One particle of asbestos in a field of view containing many thousands of other particles will be immediately apparent on scanning the eye across the field of view.

Besides optical crystallographic methods like dispersion staining, only differential thermal analysis (DTA) and X-ray diffraction have this ability to tag a particular crystalline phase in a mixture. X-ray diffraction, however, is several orders of magnitude less sensitive than dispersion staining and requires much more time. There are, however, compounds whose dispersion staining colors are, at least at first glance, confused with chrysotile colors. Here, fortunately, particle morphology is able to differentiate between these interfering substances. Quartz is one example, lizardite another. The latter, however, is a tabular talclike mineral, and quartz shows conchoidal fracture into usually thin flakes. Both are easily differentiated from fibrous asbestos by morphology.

Dispersion staining is, therefore, a straightforward technique easily applied by any microscopist with some knowledge of optical crystallography. We have found it extremely useful for the rapid and routine examination of any particulate samples for any of the various kinds of asbestos *(1)*. The necessary background information for applying the method is given in Figure 1, which plots the matching wavelength λ_0, as a function of refractive index of the Cargille refractive index liquids used in these determinations. Most of the asbestos minerals have distinctive indices without overlap. Figure 1 suggests, however, that amosite and crocidolite may overlap partially. There should be no confusion in this situation, however, since γ for amosite overlaps only with α for crocidolite. The data in Figure 1 are plotted as the averages of a considerable number of values

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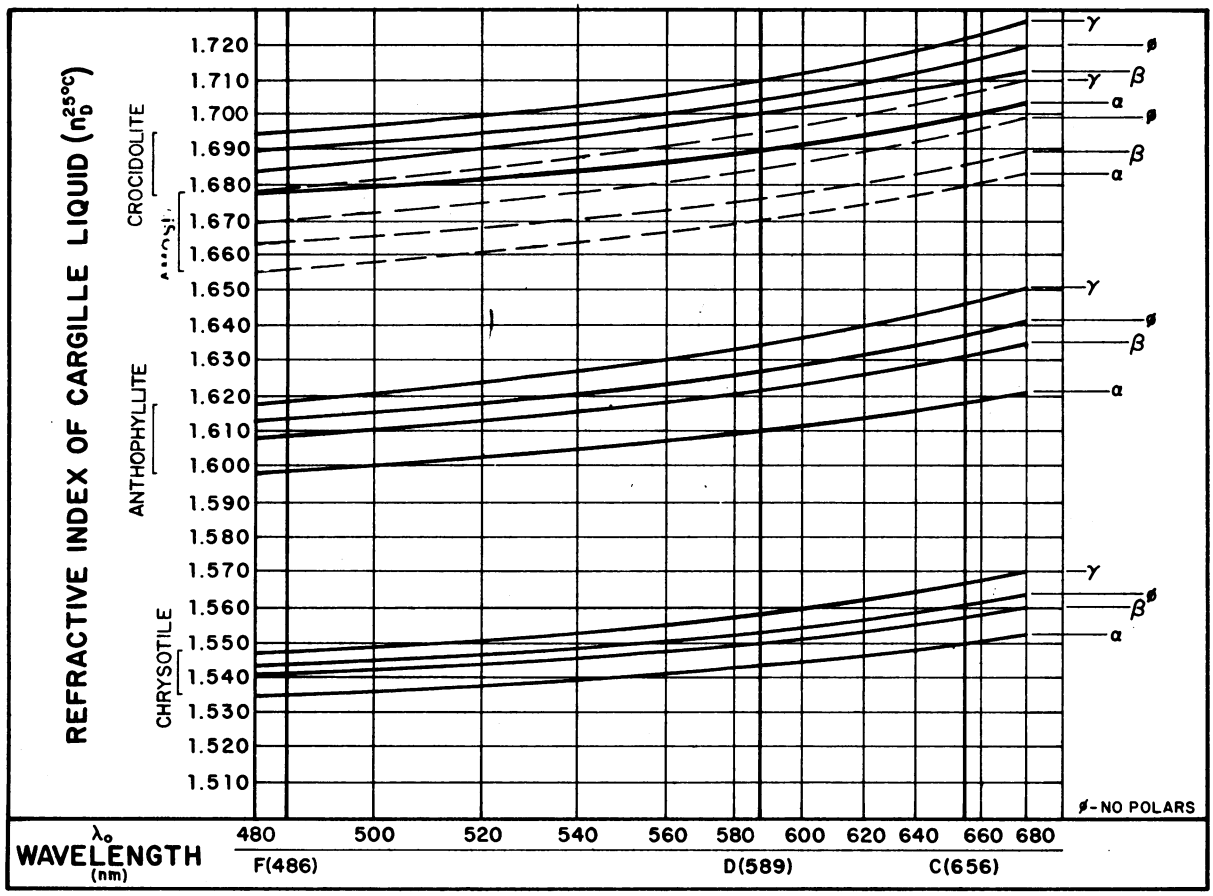

FIGURE 1. Asbestos dispersion staining curves.

for individual mine samples previously published *(1)*. It is interesting to look a little bit more closely at this variation in dispersion staining data from mine to mine, and Table 1 lists the matching wavelength λ_0 for γ (parallel to the fiber length) and α (perpendicular to the fiber length) for a group of more than 30 asbestos samples from different parts of the world. There is some variation from sample to sample, indicating composition variations. However, all of the data lie in the same characteristic chrysotile region and show no overlap with any of the fibrous amphiboles.

Since dispersion staining is a relatively new technique requiring a certain amount of skill, not only in reading the matching wavelengths but also in adjusting the microscope for best dispersion staining colors, it seems well to summarize a few of the common difficulties.

The refractive indices given in dispersion staining tables are not dispersion data for that compound. To illustrate this, we can cite the data for the ω index of quartz (Table 2).

The value 1.538 for ω at 486 nm is n_D for the Cargille liquid that matches quartz ω at 486 nm. The actual refractive index of that liquid at 486 nm is 1.550, the same as quartz ω. This operation, which simplifies the analytical procedure, is used for all dispersion staining data. One can, of course, calculate the true refractive indices of any substance from the dispersion staining data. The necessary data to do this can be found in the table of dispersion of refractive index data for the Cargille liquids.

True refractive index data and dispersion staining data are identical at 589 nm; hence, refractive index data at 589 nm for any substance become dispersion staining data for that substance. Chrysoberyl, for example, does not appear in the dispersion staining tables, but

Table 1. Matching wavelength λ_0 in H. D. 1.550 liquid.

Sample	λ_0, nm	
	$\parallel$	$\perp$
Quebec; Lake Asbestos	510	610
Quebec; King Asbestos Corp.	510	610
Quebec; Asbestos Corp.	500	610
Quebec; Bell Mines	510	600
Quebec; Johnsons	500	600
Quebec; Careys, Bradford	480	590
Quebec; Flintkote	500	610
Quebec; Normandie	570	610
Ontario; Reeves	480	590
Ontario; Munro	560	610
Vermont; Hyde Park, GAF	510	620
Vermont; Jeffrey	500	580
New Foundland; Advocate	510	610
New Foundland	590	620
Yukon; Clinton Creek	500	580
British Columbia; Cassiar	500	580
California; Pacific Asbestos Corp.	480	610
California; Coalings	590	630
Arizona	600	620
Venezuela	610	680
Rhodesia	520 (460)	620 (550)
Rhodesia; Shabina	480	580
Rhodesia; Havelock C and G	490	590
Rhodesia; Havelock HVL	490	590
Rhodesia; Havelock VRA	500	630
Cyprus	600	660
Greece; Zandini	580	620
Yugoslavia	520	590
Italian; Balengera	500 (460)	600 (510)
Russia	500	600
Australia; Woodsreef	610	680

Table 2. Refractive indices for quartz.

	486 nm	589 nm	656 nm
True refractive indices, ω	1.550	1.544	1.542
Dispersion staining data	1.538	1.544	1.547

Winchell *(2)* gives n_D = 1.746 (α), 1.748 (β), and 1.756 (γ). From these data one would mount a suspected chrysoberyl in Cargille liquid n_D = 1.750 and expect to see annular stop colors ranging from orange (β) to greenish-yellow (γ) or greenish-blue (α) to magenta-blue (γ) with the central stop. This greatly extends the usefulness of dispersion staining.

We are often asked if the dispersion staining objective can be supplied with a higher magnification. This gives us the opportunity to point out that higher magnification is not desirable. One is trying to "resolve" color of the particles not the particles themselves. Central stop dispersion staining is a darkfield procedure, hence a strong light source will show points of light for particles smaller than the resolving power limit of the microscope system, i.e., 1.22 μm for NA= 0.25, 10× objective. These points of light will be colored for particles showing dispersion in that liquid. The lower limit of detection is a function of light intensity and contrast.

There are a number of points of technique which greatly improve the sensitivity of the dispersion staining procedure. The particles must be well separated in the mounting liquid since nonstained particles close to or overlapping stained asbestos can mask their presence. The dispersion staining colors for chrysotile and the fiber amphiboles are more brilliant if one uses the high dispersion Cargille set of refractive index liquids. In spite of the above injunction concerning high magnification, it is sometimes useful to use a 20–25× ocular with the 10× dispersion staining objective. The optics for the dispersion staining objective, the axis of stage rotation, the substage apertures and lenses must be well aligned on the same optical axis. It is a good idea to take special pains to align the optical system and to maintain that microscope for dispersion staining examination only. The problem of glare from other particles in the field of view is solved to a great extent by having a centered and nearly closed field diaphragm in the optical system. This concentrates attention on particles in the center of the field and eliminates well over 90% of the glare which makes it difficult to see very fine asbestos fibers. It is desirable to be able to change the orientation of any particles which appear at first sight to give the distinctive dispersion staining colors characteristic of chrysotile (or other fibrous amphiboles). Rolling the particle by sliding the cover slip with a viscous liquid prep is the ideal way of doing this and helps greatly in differentiating quartz, paper fibers and mineral wool from chrysotile. The slides and cover slips used for dispersion staining preparations should be unusually clean since any optical discontinuities on any surface of the preparation cause glare and interfere with visibility of the dispersion staining colors.

Finally, it is desirable to have standards of the substance you are looking for mounted in the same refractive index liquid. If one is looking for chrysotile, it is also useful to have standard preparations of quartz, paper fibers, and talc in order to remind oneself quickly of the essential differences in appearance and color for these substances.

The color plates (Figs. 2–16) show the general nature of the dispersion staining colors and the specific appearance of the various kinds of asbestos in their specific liquids. Figure 2 shows the arrangement of annular and central stops in the objective back focal plane. These may be a centered 2–3 mm opening in any opaque film or a 3–4 mm dot of India ink on an 18 mm cover slip, respectively; although a dispersion staining objective is available commercially. Note that the substage iris is closed to allow only an axial beam of light to strike the object. The color series obtained with each stop are shown in Figure 3. Chrysotile is shown in Figures 4 and 5 mounted in two different standard Cargille refractive index media. Polarized light is used with an E–W vibration direction for Figure 4 and N–S for Figure 5. Observed λ_0 values are given in Table 3.

Table 3. Matching wavelengths for chrysotile.

	λ_0, nm	
Refractive index of Cargille liquid n_D	Parallel to fiber length	Crosswise to fiber
1.550	500	570
1.560	610	640

Figures 6–8 show anthophyllite all with E–W polar. Figures 6 and 7 show the fibers mounted in 1.610 and 1.620 refractive index media, respectively. Figure 8 shows two different anthophyllites (from Maryland and North Carolina) mounted in liquid of n_D 1.627. This figure, like the others, is a double exposure with the stage rotated 90° between exposures. The two different sources result in small variations in λ_0.

Amosite is shown in Figures 9–11 mounted in Cargille liquids of n_D 1.670, 1.680, and 1.690, respectively. Figure 12 shows crocidolite in Cargille liquid n_D = 1.700, and Figure 3 shows one particle of chrysotile in a talc sample mounted in Cargille liquid, n_D = 1.555.

Conclusion

The combination of particle morphology and optics uniquely identifies any of the fibrous asbestos compounds. The most rapid method for obtaining this information is through the use of dispersion staining. Properly carried out, the dispersion staining method is capable of sensitivity in the ppm range.

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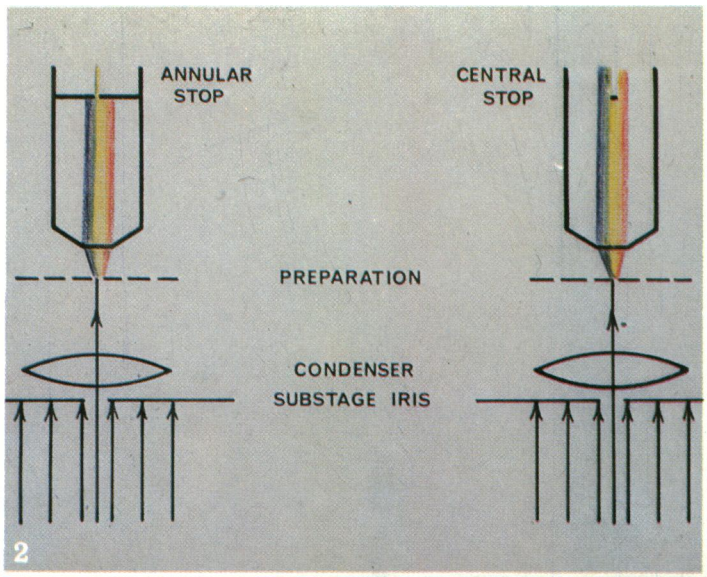

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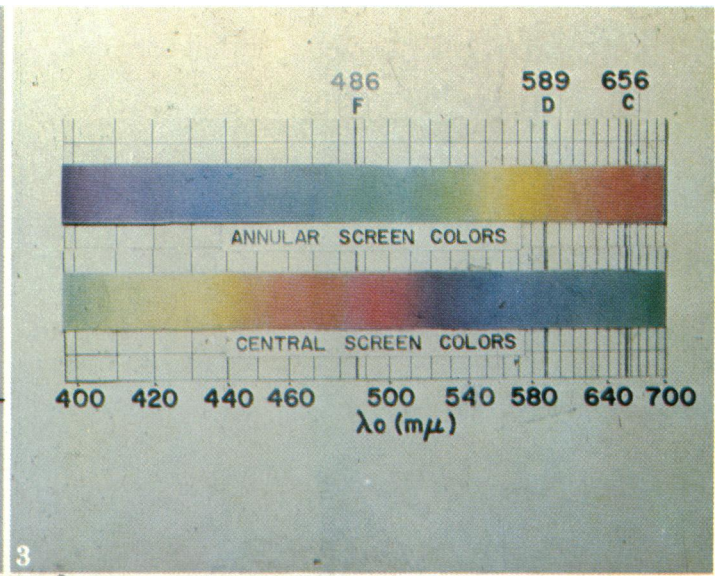

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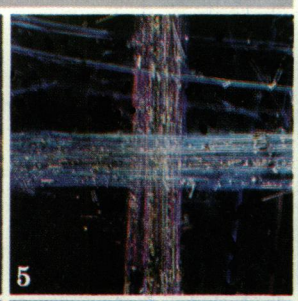
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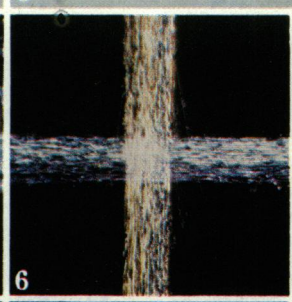
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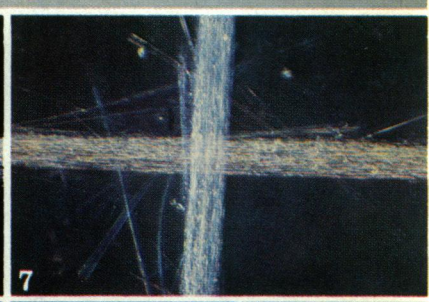
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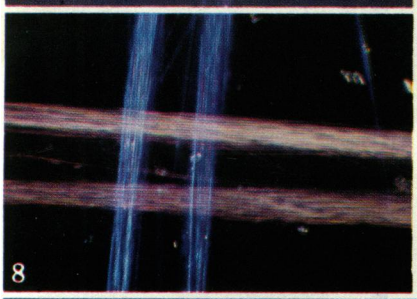
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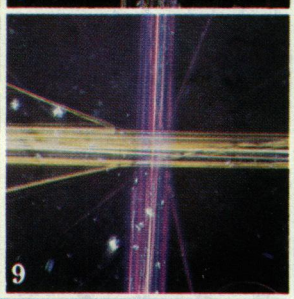
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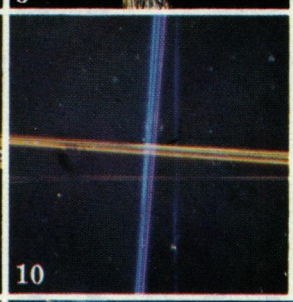
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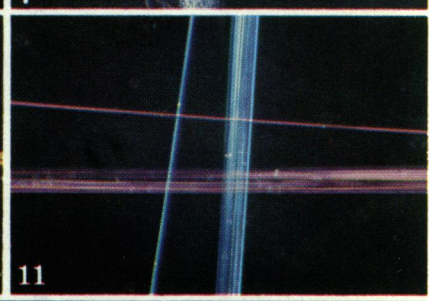
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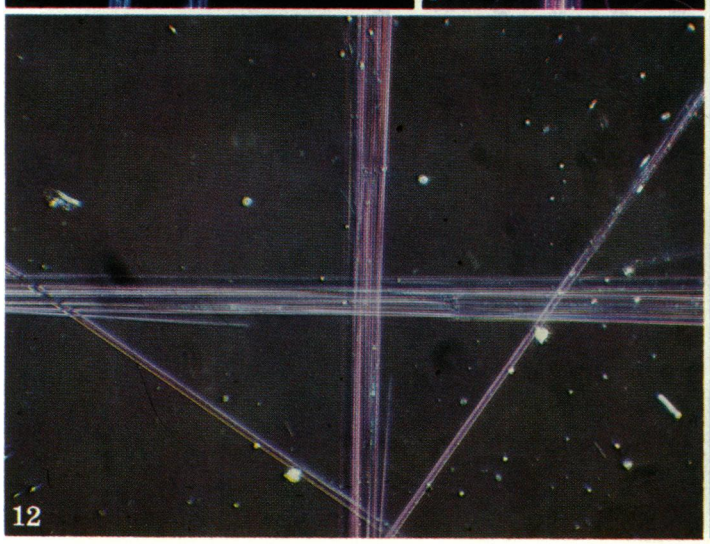
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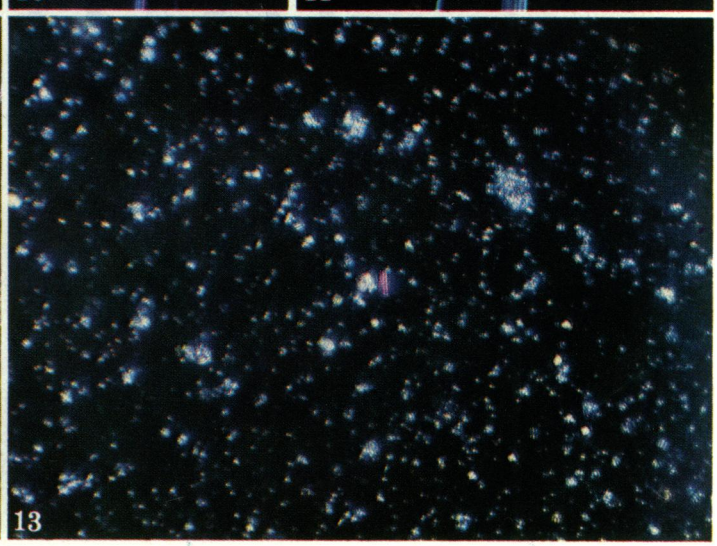
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2. Schematic arrangement for annular and central stop dispersion staining
3. Color series for annular and central stop dispersion staining
4. Chrysotile in Cargille liquid $n_D = 1.550$
5. Chrysotile in Cargille liquid $n_D = 1.560$
6. Anthophyllite in Cargille liquid $n_D = 1.610$
7. Anthophyllite in Cargille liquid $n_D = 1.620$
8. Two different anthophyllites in Cargille liquid $n_D = 1.627$
9. Amosite in Cargille liquid $n_D = 1.670$
10. Amosite in Cargille liquid $n_D = 1.680$
11. Amosite in Cargille liquid $n_D = 1.690$
12. Crocidolite in Cargille liquid $n_D = 1.700$
13. One magenta chrysotile fiber in talc mounted in Cargille liquid $n_D = 1.555$